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IS 5043 (1992): 2-Aminoanthraquinone [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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भारतीय मानक  
2-ऐमिनोऐन्थ्राक्विनान — विशिष्ट  
( दूसरा पुनरीक्षण )

*Indian Standard*

2-AMINOANTHRAQUINONE — SPECIFICATION  
( *Second Revision* )

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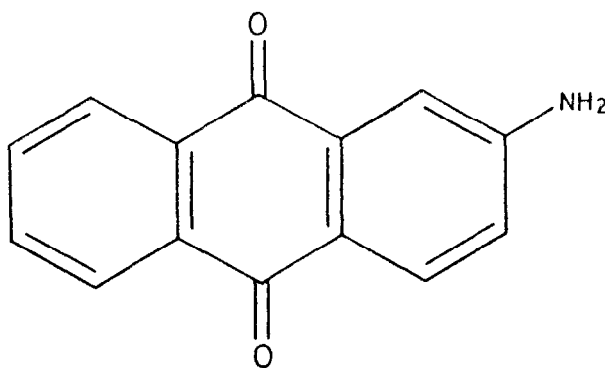
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## FOREWORD

This Indian Standard ( Second Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by Petroleum, Coal and Related Products Division Council.

2-Aminoanthraquinone (  $C_{14}H_9O_2N$  ) is an important dye intermediate used mainly in the manufacture of vat dyes. It has the following structural formula:



2-AMINOANTHRAQUINONE  
( Molecular Mass 223.2 )

This material is manufactured by aminolysis of anthraquinone-2-sulphonic acid. The commercial variety may be obtained by aminolysis of 2-chloroanthraquinone.

This standard was first published in 1969 and revised in 1976 when requirement of commercial grade of material was introduced. In the present version ( *second revision* ), the requirements of assay and sulphated ash have been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## 2-AMINOANTHRAQUINONE — SPECIFICATION

### ( Second Revision )

#### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2-aminoanthraquinone.

#### 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
1839 : 1961	Toluene, reagent grade
2552 : 1989	Steel drums (galvanized and ungalvanized) ( <i>third revision</i> )
5299 : 1969	Methods of sampling and tests for dye intermediates

#### 3 GRADES

There shall be two grades of the material, namely, Grade 1 and Grade 2.

#### 4 REQUIREMENTS

##### 4.1 Description

The material shall be in the form of orange-red crystal-

line powder and shall be free from visible impurities.

4.2 The material shall also comply with the requirements given in Table 1.

#### 5 PACKING AND MARKING

##### 5.1 Packing

The material shall be packed in steel drums (see IS 2552 : 1989) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

##### 5.2 Marking

5.2.1 Each container shall bear legibly and indelibly the following information:

- Name and grade of the material;
- Indication of the source of manufacture;
- Batch number;
- Net mass of the material; and
- Month and year of the manufacture.

5.2.2 The containers may also be marked with the Standard Mark.

#### 6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

**Table 1 Requirements for 2-Aminoanthraquinone**  
(Clauses 4.2, 6.3.1, 6.3.2 and 7.1)

Sl No.	Characteristic	Requirement		Method of Test, Ref to
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass (on dry basis), <i>Min</i>	95.0	85.0	Annex A
ii)	Moisture content, percent by mass, <i>Max</i>	0.5	0.5	Cl 9.3 of IS 5299 : 1969
iii)	Sulphated ash, percent by mass (on dry basis), <i>Max</i>	1.0	1.0	Cl 11.2 of IS 5299 : 1969
iv)	Chlorine content, percent by mass (on dry basis), <i>Max</i>	—	1.0	Annexes B & C

## 6.2 Number of Tests

**6.2.1** Test for assay shall be conducted on each of the individual samples.

**6.2.2** Tests for the determination of moisture content, sulphated ash and chlorine content shall be conducted on the composite sample.

## 6.3 Criteria for Conformity

### 6.3.1 For Individual Sample

The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfy the relevant requirement given in Table 1.

### 6.3.2 For Composite Sample

For declaring the conformity of the lot to the require-

ments of characteristics tested on the composite sample (see 6.2.2), the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

## 7 TEST METHODS

**7.1** Tests shall be carried out according to the methods prescribed in Annexes and specification as indicated in col 5 of Table 1.

### 7.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (see IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## ANNEX A [Table 1, Item (i)]

### DETERMINATION OF ASSAY

#### A-1 OUTLINE OF THE METHOD

The purity of the material is estimated by chromatographic method. 2-Aminoanthraquinone is separated chromatographically and determined quantitatively by spectrophotometer.

#### A-2 PREPARED SAMPLE

Dry the material at  $105^{\circ} \pm 1^{\circ}\text{C}$  to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests except for moisture determination.

#### A-3 APPARATUS

##### A-3.1 Chromatographic Column

A glass tube, 40 cm long, about 1.5 cm internal diameter, joined with a 50 ml thistle funnel at the upper end and fitted with a stop-cock at the lower end.

**A-3.2** Set up the column vertically. Place a cotton-wool plug in the tube and press to the bottom of the tube by means of a glass rod, flattened at the end. Place a disc of filter paper cut to the approximate internal diameter of chromatographic tube on top of the cotton wool.

**A-3.3** Prepare a slurry of about 15 g of alumina in toluene and pour it into the tube. Wash down the sides of the tube and pack the column by light tapping. Place a disc of filter paper at top surface of alumina.

**A-3.4** Always keep enough solvent in the column so that it remains wet and at least 2 cm of solvent layer

always persists over the top of alumina. On no account allow the alumina to run dry. In the event of this happening, reslurry the alumina and repack.

#### A-4 SPECTROPHOTOMETER

The readings of optical density or percentage transmittance are taken at wavelength of maximum absorption. This is obtained by plotting absorption against wavelength on a graph. The instrument shall be checked for accuracy from time to time.

#### A-5 REAGENTS

**A-5.1 Toluene** — reagent grade (see IS 1839 : 1961).

**A-5.2 Alumina** — neutral chromatographic grade. To get a material of desired activity treat it as in A-5.2.1.

**A-5.2.1** Prepare a solution containing 20 to 30 mg of pure 2-aminoanthraquinone in 100 ml of toluene. Set up a chromatographic column as in A-3.1 and chromatograph the solution. A compact main band shall be well separated from any earlier or later bands, and it shall be completely washed through the column with the addition of not less than 50 ml and not more than 150 ml of the eluent. If the chromatogram is unsatisfactory in any of these three respects and the alumina is insufficiently active, reject it. If the alumina is too active treat it as follows:

Spread out the alumina evenly on a stainless steel tray in a thin layer. Add the necessary amount of water slowly from a burette covering the whole of the tray as evenly as possible.

Mix the alumina by hand initially and then transfer to a Kilner jar and mix by rolling mechanically for at least 2 hours. Then retest for activity. If still too active, repeat the procedure adding more water in increments of 0.5 percent.

### A-5.3 2-Aminoanthraquinone — Chromatographically pure.

**A-5.3.1** Crystallize technical grade 2-aminoanthraquinone from toluene using animal charcoal for adsorption of impurities. Repeat this procedure of crystallization 3 or 4 times. The crystals obtained after final crystallization are almost pure. To make chromatographically pure material, prepare a column of 350 g of neutral alumina in toluene as in A-3.1. Dissolve 1 g of crystallized 2-aminoanthraquinone in minimum quantity of toluene and transfer it gradually to the column. Allow the solution to pass through the column and elute further by toluene. Reject the coloured bands of impurities and collect the reddish-orange band of 2-aminoanthraquinone. To get a sizeable quantity of the pure substance, repeat this procedure a few times. Concentrate the combined eluates by distillation under reduced pressure. Filter the concentrate and allow the filtrate to crystallize. Filter the pure crystals and dry them under vacuum at 100° to 105°C.

**A-5.3.2** To check the purity of the material dissolve about 60 mg, accurately weighed, in 250 ml of toluene in a volumetric flask as in A-6.1. Pass 10-ml of this solution through the alumina column in the usual manner, and dilute to 100 ml in a volumetric flask. Pipette out 10 ml of the original solution into another 100 ml volumetric flask and dilute it to the mark with the solvent. Find out the optical densities of these solutions at 410 nm. The difference between the two readings should not be more than 0.003. If the difference is more, repeat the purification procedure.

## A-6 PROCEDURE

**A-6.1** Weigh accurately about 60 mg of the prepared sample (see A-2) in a 500 ml dry conical flask. Add about 150 ml of toluene and reflux for 15 to 25 minutes. Cool and transfer the solution to a 250-ml volumetric flask. Wash the conical flask well with toluene and transfer the washings to the flask. Dilute the solution to the mark with toluene.

**A-6.2** Drain the excess solvent from the chromatographic column so that the top remains just wet.

Transfer with the help of a pipette 10 ml of the sample solution accurately and slowly to the top of the column. Add the solution to the column dropwise and at the same time drain the solvent from the column at the same rate without allowing the top of the column to go dry. Fill the upper part of the tube with solvent and allow the chromatogram to develop, keeping a good head of solvent above the alumina throughout. Reject the coloured bands preceding the main orange coloured band of 2-aminoanthraquinone.

**A-6.3** Start collecting fraction in a 100-ml volumetric flask when the main band is about 1.5 cm from the lower tip of the tube. When the entire 2-aminoanthraquinone band is eluted, remove the volumetric flask and dilute to the mark with toluene. This is the test solution.

**A-6.4** Adjust the wavelength of maximum absorption to the predetermined value and then adjust the instrument in such a manner that the transmittance through the blank becomes 100 percent after inserting the cell with blank solution. Replace the cell with the solution of the sample and read the optical density or percentage transmittance. In the case of the latter refer to the standard conversion tables and find out the corresponding optical density.

**A-6.5** Dissolve about 60 mg of accurately weighed pure substance into a 250-ml volumetric flask following the procedure for making the solution as prescribed in A-6.1. Pipette out 10 ml of this solution into another 250 ml volumetric flask and dilute it to the mark using toluene. Find out the optical density of this solution.

## A-7 CALCULATION

$$\text{Assay, percent by mass} = \frac{A}{M} \times \frac{M_1}{A_1} \times 100$$

where

$A$  = optical density of the sample;

$M_1$  = mass in mg of pure 2-aminoanthraquinone;

$M$  = mass in mg of the material taken for the test; and

$A_1$  = optical density of pure 2-aminoanthraquinone.



## ANNEX B

[Table 1, Item (iv)]

## DETERMINATION OF CHLORINE CONTENT

## B-1 OUTLINE OF THE METHOD

IKA Universal bomb, which is usually employed for micro-, semi-micro, and macro-determination of halogens and sulphur in organic compounds, is made use of to disintegrate the organically bound chlorine by fusion with sodium peroxide and ethylene glycol, thus converting it into the ionizable form. The mixture is then processed in the usual way and the halide determined by Volhard's method.

## B-2 APPARATUS

## B-2.1 IKA Universal Bomb

## B-2.2 pH Meter

## B-2.3 Silver Wire and Saturated Calomel Electrode

## B-3 REAGENTS

## B-3.1 Sodium Peroxide — granules.

## B-3.2 Ethylene Glycol — pure.

## B-3.3 Standard Silver Nitrate Solution — approximately 0.1 N.

## B-3.4 Standard Potassium Thiocyanate Solution — approximately 0.1 N.

## B-3.5 Ferric Alum Indicator Solution — 10 percent.

## B-3.6 Nitrobenzene — distilled.

## B-4 PROCEDURE

Weigh accurately about 0.3 g of the prepared sample (A-2) by difference in the tared IKA bomb. Add 7 to 8 drops of pure ethylene glycol using a dropping bottle provided with a small pipette fixed in a rubber stopper. The tip of the pipette should be sized so that 8 drops are equivalent to 160 to 170 mg of ethylene glycol. Fill up the bomb with granular sodium peroxide, adding little by little to avoid sudden reaction. The bomb should be filled up, tapping it to settle the contents to 2 mm below the edge (about 11 g of sodium peroxide), care being taken that no material is lodged between the lid and the rubber gasket. After the bomb has been filled, replace the lid and carefully close the bomb manually. Place the closed bomb in the housing provided in the safety jacket plate. The tip of the micro-flame should barely touch the bottom of the bomb. Ignition should occur after only 10 to 20 seconds (indicated by the red-hot colour at the bottom of the bomb). After further 50 seconds remove the bomb from the jacket with a pair of tongs, cool in water and open. Wash the lid thoroughly with

water and collect the washings in 500 ml beaker. Boil the fused mass along with bomb in water in the beaker covered with watch glass for half an hour and allow to cool. Acidify the solution with 25 ml of 1 : 1 sulphuric acid. Titrate potentiometrically against silver nitrate using silver wire as indicator electrode and saturated calomel as reference electrode. Note the potential difference after addition of every 0.2 ml of silver nitrate solution. The maximum potential difference indicates the end point. Continue the titration further till the difference in potential is reduced.

## B-5 CALCULATION

$$\text{Chlorine, percent by mass} = \frac{V \times N \times 3.545}{M}$$

where

$V$  = titre reading in ml;

$N$  = normality of the silver nitrate solution used; and

$M$  = mass in g of the material taken for the test.

## B-6 ALTERNATE PROCEDURE

B-6.1 Alternatively, follow the procedure as given below from the acidification stage, earlier steps remaining the same:

Acidify the solution with concentrated nitric acid and cool again. To the cold solution, add 25 ml of standard silver nitrate solution and 2 ml of nitrobenzene and shake well. Add 15 to 20 ml of 10 percent ferric alum indicator solution. Titrate with the standard potassium thiocyanate solution till the reddish-brown end point is obtained. Note this back titration reading as  $b$ . Now acidify 25 ml of silver nitrate solution with concentrated nitric acid and add 10 ml of ferric alum indicator, and titrate against standard potassium thiocyanate till the same reddish-brown end point is obtained. Note the blank titre as  $B$ .

## B-6.2 Calculation

$$\text{Chlorine, percent by mass} = \frac{(B - b) \times N \times 3.545}{M}$$

where

$B$  = titration value of blank in ml;

$b$  = titration value of test solution in ml;

$N$  = normality of standard potassium thiocyanate solution; and

$M$  = mass in g of the material taken for the test.

## ANNEX C

[Table 1, Item (iv)]

DETERMINATION OF CHLORINE BY OXYGEN FLASK METHOD  
(ALTERNATE METHOD)

## C-1 APPARATUS

C-1.1 Whatman Filter Paper No. 44 — Sheet for the preparation of paper packet.

## C-1.2 Ignition Flask

Place 25 ml distilled water in a clean 500 ml quickfit flask. Add 5 ml of 2 N sodium hydroxide solution and 0.5 ml 100 vol hydrogen peroxide. Pass oxygen gas from O<sub>2</sub> gas cylinder from 5 minutes to replace air from the flask and stopper immediately. Cover the flask with a wire gauze for safety.

## C-1.3 Wire Gauze

## C-1.4 Platinum Wire

## C-1.5 Beaker of Capacity — 500 ml.

## C-1.6 Gooch — crucible.

C-1.7 Oven — capable of maintaining temperature at  $100 \pm 5^\circ\text{C}$ .

## C-2 REAGENTS

## C-2.1 Sodium Hydroxide Solution — 2 N.

## C-2.2 Hydrogen Peroxide — 100 volume.

## C-2.3 Sulphuric Acid — 2 N.

## C-2.4 Sodium Bisulphite — 2 percent (m/v).

## C-2.5 Nitric Acid — 60 percent (m/v).

## C-2.6 Silver Nitrate Solution — N/10 (m/v).

## C-3 PROCEDURE

C-3.1 Weigh accurately by difference about 0.04 g to 0.05 g of sample (previously sieved and dried) in a

paper packet of Whatman Filter Paper No. 44 sheet previously folded using a small aluminium scoop. Place the paper packet in the gauze cup attached to platinum wire of the stopper, keeping the end of paper strip slightly projected.

C-3.2 Ignite the tip of the paper packet. When it just reaches the packet, immediately insert it into the flask and hold the stopper firmly. Cover the flask with wire gauze for safety. After ignition is over, tilt the flask upside down so that the solution seals the flask from inside. Keep the flask right down for 15 minutes. Intermittently shake the flask vigorously so as to ensure complete absorption of fumes. Remove the stopper, wash platinum wire and gauze quantitatively with few ml of distilled water into the flask. The solution is ready for estimation.

## C-3.3 Chlorine Estimation

Transfer the solution quantitatively into 500-ml beaker and boil for 5 minutes to remove peroxide, cool and neutralize with 2 N sulphuric acid (normally 5 ml required). Then add 10 ml of 2 percent sodium bisulphite solution, 10 ml of 60 percent nitric acid. Boil to expel SO<sub>2</sub> gas. Cool and filter the solution through Buchner funnel dressed with double filter paper No. 1.

Transfer the filtered solution quantitatively to 500-ml beaker and add 10 ml of N/10 AgNO<sub>3</sub>. Boil to coagulate the precipitates. Cool and filter through previously weighed Gooch crucible. Wash the precipitates to make it acid and chloride free. Then dry at  $100 \pm 5^\circ\text{C}$  to constant mass.

## C-4 CALCULATION

$$\text{C-4.1 Chlorine content, percent by mass} = \frac{M_2 \times 24.74}{M_1}$$

where

$$\begin{aligned} M_1 &= \text{mass of sample taken, and} \\ M_2 &= \text{mass of precipitates (AgCl).} \end{aligned}$$

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